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(54) Title: POWDERED COSMETIC COMPOSITION CONTAINING A FATTY SILICONE BINDER

#### (57) Abstract:

A cosmetic composition for skin consisting of an anhydrous powder and mainly including a solid particulate phase mixed with a fatty binder containing a silicone mixture which consists of (a) at least one silicone oil, (b) at least one silicone wax, (c) at least one silicone resin, (d) optionally at least one silicone gum, and (e) optionally at least one phenyldimethicone; wherein components (a), (b), (c), (d) and (e) are present in the binder in concentrations of 12-98.9%, 1-60%, 0.1-25%, 0-3% and 0-20% by weight respectively in relation to the overall weight of the silicone mixture. The composition may be used as face make-up, as eye shadow or as a make-up foundation or powder

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#### Cosmetic Composition in Powdered Form Containing a Fatty Silicone Binder

The object of this invention is a cosmetic composition for the skin, in powdered form, containing a fatty silicone binder.

It is known that some cosmetic compositions, such as rouge, eye shadow, or facial foundations or powders, are in the form of pressed or molded powders. These are anhydrous compositions called "pressed powders" (or "compacts"), consisting mainly of a mixture of powders, colored or not, and a fatty binder (oils or a mixture of oils and waxes), and shaped by compression, or by pouring into a container that serves as a mold. These powders are generally used by removing a small amount of powder and then applying it to the skin using an applicator (a sponge, a powderpuff or a brush).

The formulation of the binding agents in such pressed powders gives rise to many problems. The finished product must be sufficiently homogeneous and compact to prevent fragmentation, which is caused, in particular, by shocks, while retaining a good capacity for disintegration. Moreover, the composition must feel soft to the touch, and be easy to spread uniformly. Moreover, the binder must be compatible with the pigments, and those specializing in the art are familiar with problems related to the degradation of certain pigments when conventional fatty binders are used.

It is also known that certain make-up compositions are in the form of so-called "loose powders", in which the particles are neither compacted nor dispersed in a continuous fatty phase, but on the contrary, they retain their individuality. These loose powders often contain a fatty substance (oil) which is intended, in particular, to enhance smooth application, to promote the adherence of the powder to the skin, and to solubilize certain active ingredients. Although some loose powders may contain relatively large amounts of oil, the particles do not have a tendency to agglomerate. This is the case, in particular, with powders that contain particles in the form of hollow microspheres of a synthetic thermoplastic material; see, in particular, Patent EP-0,254,612. But the formulation of such loose powders poses the aforementioned problem of the degradation of certain pigments in the presence of conventional fatty substances.

In this application, the term "fatty binder" means a fatty substance or a mixture of fatty substances that constitutes the binder of pressed or molded powders, as well as a fatty substance or a mixture of fatty substances present in loose powders, notably to enhance smooth application and to promote adherence to the skin.

The use as binding agents, in a pressed powder, of silicone oils, which are linear polysiloxanes

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(a polydimethylsiloxane or the like, abbreviated PDMS), with low viscosity, in combination with high-viscosity PDMS (silicone gums) is described in Patent Application JP-61-180,707.

The use as binders of silicone resins (three-dimensional polycondensation products) in combination with volatile silicones is also recommended in cosmetic powders; see for example, Patent Applications JP-61-065.809, JP-61-161,211, and JP-62-298,512.

Patent Application EP-133,963 describes anhydrous cosmetic compositions containing coated pigments dispersed in a PDMS base binder, optionally in combination with a substituted linear polysiloxane (silicone wax), with a cyclic polysiloxane and/or with a conventional wax. The pigments are coated by chemical bonding with a polysiloxane to permit their dispersion in the binder.

The study of these different constituents has shown that low-viscosity silicone oils, mixed with silicone gums, are advantageous because, in particular, they provide the composition with properties of smoothness, easy spreading, and homogeneity. But the pressed make-up's durability and impact resistance properties are poor.

Polysiloxane waxes make it possible to obtain good spreading facility and acceptable make-up homogeneity, and they enhance the mechanical properties (resistance to impact). But the make-up durability properties are not satisfactory.

Silicone resins provide good durability and impact resistance properties, but the compositions lack smoothness.

It should be noted that the combination of two classes of silicone binders generally does not improve the properties appreciably.

Thus, the addition of a silicone resin to high and low-viscosity PDMS reduces spreading facility, while separately, the two constituents provide compositions that are easy to spread. In the same manner, the homogeneity provided by the two constituents individually deteriorates when they are combined. On the other hand, durability is improved.

The addition of a low-viscosity silicone oil to a silicone resin does not improve smoothness and notably reduces easy spreading and homogeneity.

It has now been discovered that the combination of three classes of silicones (oils, waxes, and resins), optionally in combination with silicone gums, to produce the fatty binder, makes it possible to obtain powders in which all of the cosmetic properties are improved or maintained at an extremely satisfactory level. In addition, this binder is compatible with all of the pigments used in cosmetic powders, including easily degradable

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inorganic pigments, such as manganese violet, or chromic oxides. Moreover, with the binder by the invention, the pigments do not need to be coated to facilitate their dispersion.

Thus, the object of this invention is a cosmetic composition for the skin in the form of an anhydrous powder, consisting principally of a solid particulate phase mixed with a fatty binder containing a silicone mixture, characterized in that said silicone mixture consists of:

- (a) at least one silicone oil,
- (b) at least one silicone wax,
- (c) at least one silicone resin,
- (d) optionally at least one silicone gum, and
- (e) optionally at least one phenyldimethicone,

and said constituents (a), (b), (c), (d), and (e) are present in the binder at concentrations of 12-98.9%, 1-60%, 0.1-25%, 0-3% and 0-20% by weight, respectively, in relation to the total weight of the silicone mixture.

The composition by the invention can be a loose powder, a pressed powder, or a molded powder.

Preferably, the concentrations of the constituents of the silicone mixture by weight in relation to the total weight of the mixture are as follows:

- pure silicone gum: 0-0.4%,

- silicone wax: 2-50%,

- pure silicone resin: 0.5-15%

- phenyldimethicone: 0-15%,

- silicone oil: qsp 100%.

In a particular embodiment, the fatty binder consists only of a mixture of silicones as defined above.

It is known that low-viscosity silicone oils are linear polysiloxanes consisting (terminal groups excepted) of units corresponding to formula (I)

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$$\begin{bmatrix} R \\ I \\ Si - O \end{bmatrix}$$

**(I)** 

in which each substituent R independently represents a lower alkyl group (with 1 to 6 carbon atoms).

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The degree of polymerization (the number of repeated structural units) of these low-viscosity polysiloxanes can range from approximately 3 to 2,000, for example.

These low-viscosity silicone oils can be prepared by known methods, or they can be purchased commercially: for example, series 47 Silbione oil (RHONE POULENC), series 200 oil (DOW CORNING), SF96 oil (GENERAL ELECTRIC).

The terminal groups are trimethylsilyl, dimethylhydroxymethylsilyl or vinyldimethylsilyl groups, for example.

Silicone gums that can be used by this invention are high molecular weight polysiloxanes which can range from 200,000 to 1,000,000, for example. They are used alone or mixed in a solvent. This solvent can be selected, in particular, from polydimethylsiloxane oils (PDMS) and polyphenylmethylsiloxane oils (PPMS). Commercial products are also known, or they can be prepared by known methods. More particularly, the following silicone gums can be cited: polydimethylsiloxane/methylvinylsiloxane, polydimethylsiloxane/diphenylsiloxane, polydimethylsiloxane/phenylmethylsiloxane, and polydimethylsiloxane/diphenylsiloxane/methylvinylsiloxane. Among commercial silicone gums, those sold under the names SE30 (GENERAL ELECTRIC), TP232 (UNION CARBIDE), Q2-1402 (DOW CORNING), or the VISCASIL series (GENERAL ELECTRIC) can be cited.

Silicone waxes that can be used in the fatty binder of this invention are substituted polysiloxanes that are solid or liquid at room temperature. They are preferably liquids or solids with a low melting point. In particular, they are substituted linear polysiloxanes consisting essentially (aside from the terminal groups) of structural units corresponding to formulas II and III, in the respective molar ratios of m and n:

$$\begin{bmatrix}
R \\
| \\
Si - O
\end{bmatrix}_{m} \quad \text{and} \quad \begin{bmatrix}
R' \\
| \\
Si - O
\end{bmatrix}_{n}$$
(II)

in which each substituent R is defined as above,

each R' independently represents alkyl (linear or branched), and optionally unsaturated, with 6-30 carbon

atoms, or an -X-R" group,

each X independently represents:

-0-,

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-(CH<sub>2</sub>)<sub>3</sub>-O-CO-,

-(CH<sub>2</sub>)<sub>b</sub>-CO-O-,

a and b independently represent numbers between 0 and 6, and

each R" independently represents an alkyl group, optionally unsaturated, with 6-30 carbon atoms,

m is a number between 0 and 400, and in particular, from 0 to 100,

n is a number between 1 and 200, and in particular, from 1 to 100,

while the total (m + n) is less than 400, and in particular less than or equal to 100.

These silicone waxes are known, or they can be prepared by known methods. Among this type of commercial silicone waxes, those sold under the names of Abilwax 9800, 9801 or 9810 (GOLDSCHMIDT), KF910 and KF7002 (SHIN ETSU), or 176-1118-3 and 176-11481 (GENERAL ELECTRIC) can be cited.

Silicone waxes that can be used can also be selected from formula IV compounds

$$R_1$$
-Si(CH<sub>3</sub>)<sub>2</sub>-O-[Si(R)<sub>2</sub>-O-]<sub>z</sub>-Si(CH<sub>3</sub>)<sub>2</sub>-R<sub>2</sub> (IV)

in which R is defined as above, and  $R_1$  represents an alkyl group with 1-30 carbon atoms, an alkoxy group with 6 to 30 carbon atoms, or a group with the formula:

O O 
$$\parallel$$
  $\parallel$   $-$  (CH<sub>2</sub>)<sub>a</sub>-C-O-R" or  $-$  (CH<sub>2</sub>)<sub>b</sub>-O-C-R"

R<sub>2</sub> represents an alkyl group with 6-30 C, an alkoxy group with 6 to 30 C or a group with the formula:

O O 
$$\parallel$$

$$-(CH2)a-C-O-R"$$
Or  $-(CH2)b-O-C-R"$ 

wherein a and b represent a number from 0 to 6.

R" is a C<sub>6</sub> to C<sub>30</sub> alkyl,

and z is a number between 1 and 100.

Among the formula IV silicone waxes that are known products or that can be prepared by known

methods, the following commercial products can be cited, in particular: Abilwax 2428, 2434, and 2440 (GOLDSCHMIDT), or VP 1622 and VP 1621 (WACKER).

Silicone resins are the hydrolysis and polycondensation products of siloxane mixtures with the formulas (R), SiOCH<sub>3</sub> and Si(OCH<sub>3</sub>)<sub>4</sub>, with R representing an alkyl group with 1-6 carbon atoms.

These silicone resins are known or can be prepared by known methods. Among the commercial silicone resins that can be used, those sold under the names DC 593 (DOW CORNING) or SS 4230 (GENERAL ELECTRIC) can be cited as examples.

Phenyldimethicones are known products that correspond to formula VI:

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$$(R^{\prime\prime\prime})_3\text{-Si-O} = \begin{bmatrix} R^{\prime\prime\prime} \\ \vdots \\ \text{Si-O} \\ C_6H_5 \end{bmatrix}_p \begin{bmatrix} \text{CH}_3 \\ \vdots \\ \text{Si-O} \\ \text{CH}_3 \end{bmatrix}_q \text{ (VI)}$$

in which

q is a number between 0 and 5,000,

p is a number between 1 and 5,000,

and each R" independently represents a methyl, phenyl, or trimethylsilyloxy group.

These phenyldimethicones can be used as optional ingredients to enhance smooth application.

Generally, the fatty binder can represent 0.5 to 25% by weight, preferably 3 to 20%, of the total weight of the composition.

The particulate phase of the composition consists of the pigments and/or fillers which are generally used in such cosmetic compositions. The pigments are selected from inorganic and/or organic pigments and/or nacreous pigments.

These pigments can represent up to 70% by weight of the final composition.

Among inorganic pigments, the following can be cited as examples:

- titanium dioxide (rutile or anatase), optionally surface treated, and coded in the Color Index under reference number CI 77891;
  - black, yellow, red and brown iron oxides, coded under reference numbers CI 77499, 77492, 77491;
  - manganese violet (CI 77742);
  - ultramarine violet (CI 77007);

- ultramarine blue (CI 77007);
- chromium oxide (CI 77288);
- hydrated chromic oxide (CI 77289) and
- ferric blue (CI 77510).
- 5 Among organic pigments, the following can be cited, in particular:
  - D&C red No. 3 (CI 45430:1)
  - D&C red No. 6 (CI 15850:2)
  - D&C red No. 7 (CI 15850:1)
  - D&C red No. 9 (CI 15585:1)
- 10 D&C red No. 13 (CI 15630:3)
  - D&C red No. 19 (CI 45170)
  - D&C red No. 21 (CI 45380:2)
  - D&C red No. 27 (CI 45410:1)
  - D&C red No. 30 (CI 73360)
- 15 D&C red No. 36 (CI 12085),
  - carbon black (CI 77266) and cochineal carmine (CI 75470).

Nacreous pigments can be selected, in particular, from white nacreous pigments, such as mica coated with titanium oxide or bismuth oxychloride. Colored nacreous pigments can also be used, such as titanium mica colored with iron oxides, titanium mica colored with ferric blue or chromic oxide, titanium mica colored with an organic pigment of the aforementioned type, as well as bismuth oxychloride base nacreous pigments.

Fillers are selected, in particular, from:

- Talc, which is a hydrous magnesium silicate used in the form of particles, generally with a particle size of less than 40  $\mu$ m; talc has moisture absorbing properties and is used mostly because of its oily feel;
- micas, which are aluminosilicates with various compositions, are in the form of scales in dimensions of 2 to 200  $\mu$ m, preferably 5 to 70  $\mu$ m, and a thickness of 0.1 to 5  $\mu$ m, preferably 0.2 to 3  $\mu$ m. Micas can be natural (for example, muscovite, margarite, roscoelite, lipidolite, and biotite) or synthetic. Micas are generally transparent and give the skin a satiny appearance;
  - starch, modified or not, in particular rice starch;
  - silicon dioxide;
- 30 aluminum oxide;

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- boron nitride;
- kaolin, which is a hydrated aluminum silicate that is present in the form of isotropic particles, and has good fatty substance absorption properties;
- zinc and titanium oxides: these oxides have an oily feel, good covering power, and significant opacity; the nanopigmentary forms of these products can also be used;
- precipitated calcium carbonate, which is in the form of particles of less than approximately 10  $\mu$ m, has an oily feel and produces a matte appearance;
  - magnesium carbonate or bicarbonate, which has, in particular, perfume fixing properties;
- metallic soaps derived from organic carboxylic acids with 8 to 22 carbon atoms, preferably 12 to 18 carbon atoms, for example zinc, magnesium, or lithium stearate, zinc laurate, magnesium myristate, etc. These soaps, which are generally in the form of particles of less than 10  $\mu$ m, have an oily feel and help the powder to adhere to the skin;
- synthetic powdered polymers (or copolymers) selected from polyethylene and its derivatives (e.g.: polytetrafluoroethylene, polystyrene, etc.), polyacrylates, polymethacrylates, polyesters, or polyamides, etc., for example, nylon powder.
- powders in the form of hollow microspheres made of a synthetic thermoplastic material, in which the hollow part contains a gas.

Hollow microspheres are prepared by known processes, such as described in Patent FR 3,615,972 or European Patent Application No. 0,056,219.

These microspheres can be made from any non-toxic and non-irritating thermoplastic materials. For example, these materials can be polymers or copolymers of ethylene derivatives (for example, polyethylene, polystyrene, vinyl chloride-acrylonitrile copolymer, etc.), polyesters, urea-formaldehyde polymers, vinylidene chloride copolymers (for example, vinylidene chloride-acrylonitrile), etc.

Fillers can represent up to 95% of the total weight of the composition of the invention.

Pigments and fillers can be coated, if desired, with substances such as, in particular, amino acids, silicones, metallic soaps, or collagen; notably to modify their surface condition. The surface condition can also be modified by chemical grafting or adsorption of silicone molecules, other molecules may also be suitable, such as triisostearoyl titanate.

Several common additives can also be introduced into the composition, and taken altogether, these additives generally represent no more than 10%, and in particular, no more than 5% by weight of the total

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weight of the composition. The composition of the invention can thus contain at least one additive selected, in particular, from antiseptics (for example, trichlorodiphenyl ether, cationic agents, boric acid, etc.) which are used, in particular, in deodorant powders for the body or feet and in baby powders; astringent agents, which are used in deodorant powders or in foot powders, such as aluminum hydroxychloride or alums; sun screens; healing agents; anti-free radical agents; vitamins; softening agents; emollient agents, in particular, oils such as fatty acid esters with a  $C_{10}$  to  $C_{22}$  fatty alcohol or a lower alcohol (for example, triisocetyl citrate, myristyl myristate, etc.) or vegetable oils (in particular, jojoba oil, etc.), mineral oils (in particular, vaseline oil, etc.) or animal oils (in particular, lanolin, etc.); hydrating agents (glycerol, sorbitol, etc.); depigmenting agents; perfumes; thickening agents (natural or synthetic gums); etc.

In particular, the compositions of the invention can be in the form of rouge, eye shadow, face powders, body powders (perfumed and/or deodorant) including foot powders, etc.

The compositions of the invention can be prepared by the usual methods, for example, by one of the following methods:

#### Method 1: (for pressed powders)

First, pigments and/or fillers are mixed, as well as powdered additives, then the binder and/or any thickening agents, as well as any other ingredients are added, and they are all blended and/or possibly pulverized.

The binder can be heated if necessary.

A press is then used to compact the mixture in metallic cupels.

Method 2: (for molded powders)

All constituents of the formula are mixed and suspended in a solvent (water, hexane, isopropanol, ethanol, etc.).

The paste obtained is then poured into a cupel, and the solvent is evaporated.

Method 3: (for loose powders)

Pigments and/or fillers, as well as powdered additives are mixed, then the binder, and optionally thickening agents, as well as any other ingredients are added, and mixed together and optionally pulverized.

If necessary, the binder is heated. If desired, the mixture can be screened before packaging in a suitable container.

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Another object of the invention is the use of a silicone mixture as a binding agent, in the preparation of an anhydrous cosmetic composition in powder form (loose, pressed, or molded) based principally on solid particles mixed with a fatty binder, said silicone mixture being as defined above. The compositions obtained are applied to the skin by the usual methods.

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The following examples illustrate the invention. In these examples, the quantities of the different ingredients are given in parts by weight.

#### EXAMPLE 1: Rouge

| 15 |  |        |
|----|--|--------|
|    | Part A   |        |
|    | Titanium dioxide                               | 10.0   |
|    | Titanium mica                                  | 10.00  |
|    | Zinc stearate                                  | 4.00   |
| 20 | D&C Red 30                                     | 0.50   |
|    | Talc   | 69.30  |
|    |  |        |
|    | Рап В  |        |
|    | Cetyldimethicone                               | 0.08   |
| 25 | Behenoxydimethicone                            | 1.60   |
|    | 33% Trimethylsiloxysilicate in                 |        |
|    | low-viscosity PDMS                             | 0.53   |
|    | 14% High-viscosity polydimethylsiloxane active |        |
|    | substance in a low-viscosity PDMS base         | 0.01   |
| 30 | Low-viscosity polydimethylsiloxane             | 3.78   |
|    | Preservatives                                  | 0.2    |
|    |  |        |
|    |  | 100.00 |

100.00

Source of products:

Cetyldimethicone: Abilwax 9801 (GOLDSCHMIDT)

Behenoxydimethicone: Abilwax 2440 (GOLDSCHMIDT)

33% Trimethylsiloxysilicate in a low-viscosity PDMS; DC593 (DOW CORNING).

High-viscosity polydimethylsiloxane: Q2-1403 (DOW CORNING): 14% solution in a low-viscosity PDMS.

Low-viscosity polydimethylsiloxane: PDMS, 10 centistokes, sold by the GOLDSCHMIDT Company.

#### Operating Method:

- 1) Mix phase A constituents.
  - 2) Add phase B. Mix again
  - 3) Optionally pulverize
  - 4) Screen
  - 5) Press into metallic cupel.

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This formula can be pulverized very readily and it provides a very homogeneous make-up.

#### **EXAMPLE 2**: Pressed face powder

| 20 | Part A                             |        |
|----|------------------------------------|--------|
|    | Sericite                           | 65.80  |
|    | Mica                               | 15.00  |
|    | Polyethylene powder                | 5.00   |
|    | Titanium dioxide                   | 2.00   |
| 25 | Iron oxides                        | 8.00   |
|    | Part B                             |        |
|    | Cetyldimethicone                   | 0.15   |
|    | Behenoxydimethicone                | 0.30   |
|    | Trimethylsiloxysilicate (DC 593)   | 0.75   |
| 30 | Low-viscosity polydimethylsiloxane | 2.80   |
|    | Preservatives                      | 0.2    |
|    |                                    | 100.00 |

Source of Products: See Example 1

Operating Method: same as Example 1.

This product provides smooth application.

#### 5 <u>EXAMPLE 3</u>: Eye shadow

|    | Part A                             |        |
|----|------------------------------------|--------|
|    | Titanium mica                      | 40.00  |
|    | Green chromic oxide                | 6.00   |
| 10 | Talc                               | 43.80  |
|    | Part B                             |        |
|    | Cetyldimethicone                   | 1.24   |
|    | Trimethylsiloxysilicate (DC593)    | 1.98   |
|    | Low-viscosity polydimethylsiloxane | 6.78   |
| 15 | Preservative                       | 0.20   |
|    |                                    |        |
|    |                                    | 100.00 |
|    | C. Ethanol                         | 85.00  |

20 Source of Products: See Example 1

Operating Method:

- 1) Mix phase A constituents
- 25 2) Add phase B and phase C and mix again.
  - 3) The paste obtained is poured or injected directly into a cupel
  - 4) The solvent is evaporated.

This product is extremely easy to apply and it is very smooth.

#### EXAMPLE 4: Eye shadow

|    | Part A                             |        |
|----|------------------------------------|--------|
|    | Mica                               | 15.00  |
| 5  | Powdered polyamide                 | 10.00  |
|    | Iron oxide                         | 10.00  |
|    | Manganese violet                   | 20.00  |
|    | Talc                               | 38.80  |
|    | Part B                             |        |
| 10 | Cetyldimethicone                   | 0.21   |
|    | Behenoxydimethicone                | 0.44   |
|    | Trimethylsiloxysilicate (DC 593)   | 1.12   |
|    | Low-viscosity polydimethylsiloxane | 4.23   |
|    | Preservatives                      | 0.20   |
| 15 |                                    |        |
|    |                                    | 100.00 |
|    |                                    |        |

Source of Products: See Example 1

#### Operating Method:

20 1) Mix phase A constituents

- 2) Add phase B and mix again
- 3) Optionally pulverize
- 4) Screen
- 5) Press into a metallic cupel

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This product is very easy to apply and it is very smooth.

#### **EXAMPLE 5**: Loose powder

| 30 | Part A                                       |       |
|----|--|-------|
|    | Mica   | 67.50 |
|    | Powdered polyamide                           | 25.00 |
|    | Zinc stearate                                | 2.00  |
|    | Iron oxides                                  | 1.00  |
| 35 | Perfume (impregnated in magnesium carbonate) | 1.5   |

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|                    | 100.00 |
|--------------------|--------|
| Low-viscosity PDMS | 1.0    |
| DC593              | 0.5    |
| Abilwax 9801       | 1.5    |
| Part B             |        |

Low-viscosity PDMS is sold under the name Abil 10 (GOLDSCHMIDT).

10 Operating Method:

- 1) Mix part A constituents.
- 2) Add part B and mix again
- 3) Pulverize and screen.

#### 15 COMPARATIVE EXAMPLES

The properties of the pressed powders obtained were studied using different silicones or silicone mixtures as binding agents. In all cases, the particulate phase was the same as in Example 4(A) above, and the proportion of the binding agent was 6%. The compositions were prepared as in Example 4.

The compositions obtained were tested by users who had to indicate, for the property tested, whether the test composition gave an average result (0), a good result (+), or a very good result (++),

The following properties were investigated:

- a) The mechanical properties of the pressed powder
- resistance to impact: the test consisted of evaluating the cohesion of the pressed product by measuring the loss of powder mass after 10 standardized drops from a height of 20 cm,
  - b) Cosmetic Properties:
  - Smooth application: the test consisted of evaluating (sensory evaluation) the smoothness of application;
- ease of spreading: the test consisted of evaluating how easily the powder spreads and can be applied over the entire surface to be made up;
- adherence: the test consisted of evaluating the powder's capacity to be applied and to remain in place on the skin;

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- durability: the test consisted of evaluating the powder's ability to remain on the skin 4 hours after it was applied;

- homogeneity: the test consisted of evaluating the uniformity of the powder layer on the skin after make-up is applied.

The compositions investigated and the results are summarized in Tables 1 and 2. The compositions studied in Table 2 are compositions by the invention, while the compositions in Table 1 are comparison compositions.

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TABLE 1
Comparison Compositions

| COMPOSITIONS              | -    | 2    | 3    | 4    | 5    | 9   | 7   | 8   | 6   |
|---------------------------|------|------|------|------|------|-----|-----|-----|-----|
| LOW-VISCOSITY PDMS        | %001 |      |      |      |      | %09 | 30% | 20% | 20% |
| HIGH-VISCOSITY PDMS*      |      | 100% |      |      |      | 40% | 20% |     |     |
| BEHENOXYDIMETHICONE       |      |      | 100% |      |      |     |     | 20% |     |
| CETYLDIMETHICONE          |      |      |      | 100% |      |     |     |     |     |
| TRIMETHYLSILOXYSILICATE   |      |      |      |      | %001 |     | 20% |     | 20% |
| PROPERTIES                |      |      |      |      |      |     |     |     |     |
| Resistance to impact      | 0    | 0    | ++   | +    | ++   | 0   | 0   | +   | +   |
| Smoothness of application | ++   | 0    | +    | +    | 0    | +   | +   | ++  | 0   |
| Have of spreading         | ++   | +    | ++   | +    | +    | +   | +   | +   | 0   |
| Durability                | +    | 0    | 0    | 0    | ++   | 0   | ++  | 0   | ++  |
| Homoveneity               | ++   | +    | +    | +++  | +    | + + | 0   | ++  | 0   |
| Adherence                 | 0    | ++   | +    | +    | +    | +   | +++ | 0   | +   |
|                           |      |      |      |      |      |     |     |     |     |

Source of Products: See Example 1

\*14% solution of active substance dissolved in a low-viscosity PDMS.

TABLE 2

Compositions No. 10-15 According to the Invention

| 5  | COMPOSITIONS             | 10   | 11   | 12   | 13   | 14   | 15   |
|----|--------------------------|------|------|------|------|------|------|
|    | CETYLDIMETHICONE         | 3.8  | 1.3  | 12.4 | 20.0 | 20.8 | 2.0  |
|    | BEHENOXYDIMETHICONE      | 7.3  | 26.7 | -    | 20.0 | 0.5  | 25.0 |
|    | TRIMETHYLSILOXYSILICATE* | 18.7 | 8.8  | 19.8 | 6.6  | 24.7 | 3.0  |
|    | HIGH-VISCOSITY PDMS**    | -    | 0.2  | -    | 0.1  | -    | 1.0  |
| 10 | LOW-VISCOSITY PDMS       | 70.2 | 63.0 | 67.8 | 53.3 | 54.0 | 69.0 |
| !  | PROPERTIES               |      |      |      |      |      |      |
|    | Resistance to impact     | +    | +    | +    | ++   | ++   | +    |
|    | Smooth application       | ++   | ++   | ++   | +    | +    | ++   |
|    | Ease of spreading        | +    | +    | ++   | ++   | +    | ++   |
| 15 | Durability               | +    | +    | ++   | +    | ++   | +    |
|    | Homogeneity              | +    | ++   | ++   | +    | ++   | ++   |
|    | Adherence                | ++   | +    | +    | ++   | +    | +    |

<sup>\* 33%</sup> active substance in a low-viscosity PDMS

Source of Products: See Example 1

#### 2) Storage Results:

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Compositions No. 10, 11 and 12 above were compared with a composition containing a conventional binder and the same particulate phase. The compositions were stored in an oven (40°C) for 2 months.

At the end of this time, the intensity of the odor was evaluated subjectively, and scored as follows:

- very intense: +++
- intense: ++
- fairly intense: +
  - no odor: 0

The conventional compositions contained the following binder:

<sup>\*\* 14%</sup> active substance in a low-viscosity PDMS

#### **COMPOSITION 16:**

- vaseline oil: 55%

- oleic alcohol: 30%

- liquid lanolin: 10%

- castor oil: 5%

#### **COMPOSITION 17:**

- vaseline oil: 85%

- white vaseline: 15%

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The results are summarized in Table 3 below:

#### TABLE 3

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| COMPOSITIONS | 10 | 11 | 12 | 16  | 17 |
|--------------|----|----|----|-----|----|
| Odor         | 0  | 0  | 0  | +++ | +  |

#### 3) Preparing Compositions Containing Pigments that are Difficult to Use

It is known that certain pigments, such as chromic oxide, ultramarine blue, and manganese violet lead to formulations that are difficult to use and are not very homogeneous in make-up.

The following compositions were tested:

- talc: 40%

- chromic oxides: 15%

- mica: 20%

- titanium mica coated with chromic oxide: 15%

- binder: 10%

The chromic oxide used is a 50/50 mixture of chromic oxide and chromium hydroxide.

The following binding agents were used for test compositions 18, 19, and 20:

- Composition 18: binder of composition 16

- Composition 19: binder of composition 11 (according to the invention)
- Composition 20: binder of composition 12 (according to the invention)

The results are summarized in the following table (Table 4):

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TABLE 4

10

 TEST COMPOSITIONS
 18
 19
 20

 Smoothness
 0
 +
 +

 Ease of spreading
 +
 ++
 ++

 Homogeneity
 0
 +
 ++

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#### **CLAIMS**

1. Cosmetic composition for the skin in the form of an anhydrous powder containing principally a solid particulate phase mixed with a fatty binder containing a silicone mixture, characterized in that said silicone mixture consisted of:

- (a) at least one silicone oil,
- (b) at least one silicone wax,
- (c) at least one silicone resin,
- (d) optionally at least one silicone gum, and
- (e) optionally at least one phenyldimethicone,

and that said constituents (a), (b), (c), (d) and (e) are present in the binder at concentrations of 12-98.9%, 1-60%, 0.1-25%, 0-3%, and 0-20% by weight in relation to the total weight of the silicone mixture.

2. Composition according to the preceding claim, characterized in that the concentrations of the constituents of the silicone mixture, by weight in relation to the total weight of said mixture are as follows:

- silicone wax:

2-50%,

- silicone resin:

0.5 - 15%,

- silicone gum:

0-0.4%,

- phenyldimethicone:

0-15%,

- silicone oil: qsp 100%.

- 3. Composition according to any one of the preceding claims, characterized in that said fatty binder consists of said silicone mixture.
- 4. Composition according to any one of the preceding claims, characterized in that the low-viscosity silicone oil is at least a linear polysiloxane consisting (terminal groups excepted) of formula I structural units

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$$\begin{bmatrix}
R \\
| \\
Si-O
\end{bmatrix}$$
R

30

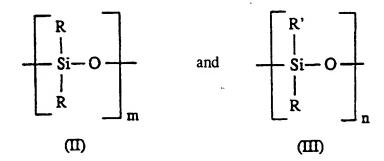
**(I)** 

in which each R substituent independently represents a lower alkyl group with 1-6 carbon atoms.

with the polysiloxane having a degree of polymerization of 3 to 2,000.

5. Composition according to any one of the preceding claims, characterized in that the silicone wax consists essentially of at least one substituted linear polysiloxane consisting essentially (aside from the terminal groups) of structural units of formulas (II) and (III), in molar proportions m and n, respectively:

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in which each substituent R is an alkyl group with 1 to 6 carbon atoms.

each R' independently represents an alkyl, optionally unsaturated, having 6 to 30 carbon atoms, or an -X-R" group;

each X independently represents:

-0-

20 -(CH<sub>2</sub>)<sub>a</sub>-O-CO-,

-(CH<sub>2</sub>)<sub>b</sub>-CO-O-,

a and b independently represent numbers between 0 and 6, and

each R" independently represents an alkyl group, optionally unsaturated, with 6 to 30 carbon atoms,

m is a number between 0 and 400, and in particular, from 0 to 100,

n is a number between 1 and 200, and in particular, from 1 to 100,

the total (m + n) is less than 400, and in particular, less than or equal to 100.

6. Composition according to any one of the preceding claims, characterized in that said silicone wax contains at least one formula IV compound:

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$$R_1$$
-Si(CH<sub>3</sub>)<sub>2</sub>-O-[Si(R)<sub>2</sub>-O-]<sub>z</sub>-Si(CH<sub>3</sub>)<sub>2</sub>-R<sub>2</sub> (IV)

in which each R is an alkyl group with 1 to 6 carbon atoms,

 $R_1$  represents an alkyl group with 1 to 30 carbon atoms, an alkoxy group with 6 to 30 carbon atoms, or a group:

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O O 
$$\| - (CH_2)_{a}$$
-C-O-R" or  $- (CH_2)_{b}$ -O-C-R'

R, represents an alkyl group with 6 to 30 C atoms, an alkoxy group with 6 to 30 C atoms or a group:

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O O 
$$\|$$
 Or  $-(CH_2)_a$ -C-O-R"  $-(CH_2)_b$ -O-C-R"

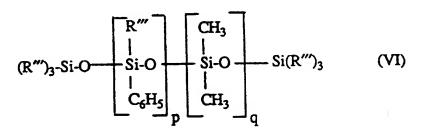
a and b represent a number from 0 to 6,

R" is a C<sub>6</sub> to C<sub>30</sub> alkyl,

and z is a number between 1 and 100.

- 7. Composition according to any one of the preceding claims, characterized in that said silicone resin consists of the hydrolysis and polycondensation products of siloxane mixtures with the formula (R)<sub>3</sub>SiOCH<sub>3</sub> and Si(OCH<sub>3</sub>)<sub>4</sub>, with R representing an alkyl group with 1 to 6 carbon atoms.
- 8. Composition according to any one of the preceding claims, characterized in that said silicone gum is a polysiloxane with a molecular weight of 200,000 to 1 million.
- 9. Composition according to any one of the preceding claims, characterized in that said phenyldimethicone corresponds to formula VI.

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in which

q is a number between 0 and 5,000,

p is a number between 1 and 5,000,

and each R'" independently represents a methyl, phenyl, or trimethylsilyloxy group.

10. Composition according to any one of the preceding claims, characterized in that said silicone mixture represents 0.5 to 25%, and in particular 3 to 20% by weight in relation to the total weight of the composition.

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- 11. Composition according to any one of the preceding claims, characterized in that it also includes at least one additive agent selected from antiseptics, astringent agents, sun screens, healing agents, anti-free radical agents, vitamins, softening agents, emollient agents, depigmenting agents, perfumes, and thickening agents.
- 12. Composition according to the preceding claim, characterized in that the additives, taken together, represent no more than 10%, and in particular, no more than 5%, of the total weight of the composition.
- 13. Composition according to any one of the preceding claims, characterized in that it is in the form of an eye shadow, a rouge, a facial powder, or a body powder.
- 14. Use of a silicone mixture in the preparation of an anhydrous cosmetic composition in the form of a powder consisting of solid particles mixed with a fatty binder containing a silicone mixture, said silicone mixture consisting of:
  - (a) at least one silicone oil,
  - (b) at least one silicone wax,
  - (c) at least one silicone resin,
  - (d) optionally at least one silicone gum, and
- (e) optionally at least one phenyldimethicone,

said constituents (a), (b), (c), (d), and (e) being present in the binder at concentrations of 12-98.9%, 1-60%, 0.1-25%, 0-3%, and 0-20%, respectively, of the total weight of said silicone mixture.

15. Use according to the preceding claim, characterized in that said composition and/or said silicones are as defined in any one of Claims 2 to 12.

[circled pp. 26-31 - French and English Search Reports]

#### INTERNATIONAL SEARCH REPORT

International application No. PCT/FR 93/00221

| A. CLASSIFICATION OF SUBJECT MATTER    |  |  |   |  |  |
|--|--|--|---|--|--|
| Int.Cl                                 | .5 A61K7/48; A61K7/035   |  | ·   |  |  |
| According t                            | o International Patent Classification (IPC) or to both   | national classification and IPC  |   |  |  |
|  | DS SEARCHED  |  |   |  |  |
|  | cumentation searched (classification system followed by  | classification symbols)  |   |  |  |
| Int.Cl                                 | .5 A61K  |  |   |  |  |
| Documentati                            | on searched other than minimum documentation to the ex   | stent that such documents are included in th   | e fields searched                                     |  |  |
| Electronic de                          | ts base consulted during the international search (name o  | data base and, where practicable, search t   | erms used)  |  |  |
| C. DOCUMENTS CONSIDERED TO BE RELEVANT |  |  |   |  |  |
| Category*                              | Citation of document, with indication, where ap  | propriate, of the relevant passages  | Relevant to claim No.                                 |  |  |
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|  |  | ,  |   |  |  |
|  |  | -/   |   |  |  |
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| Further                                | er documents are listed in the continuation of Box C.  | See patent family annex.   | ·   |  |  |
| "A" docume                             | categories of cited documents:<br>at defining the general state of the art which is not considered<br>particular relevance   | "I" later document published after the inte<br>date and not in conflict with the appli<br>the principle or theory underlying the                         | ication but cited to understand                       |  |  |
| "E" earlier o                          | locument but published on or after the international filing date<br>at which may throw doubts on priority claim(s) or which is<br>establish the publication date of another ciution or other   |  | dered to involve an inventive                         |  |  |
| special "O" docume means               | reason (as specified) nat referring to an oral disclosure, use, exhibition or other  | "Y" document of particular relevance; the considered to involve an inventive combined with one or more other such being obvious to a person skilled in a | step when the document is documents, such combination |  |  |
|  | nt published prior to the international filing date but later than<br>rity date claimed  | "&" document member of the same pater  | t family  |  |  |
|  | June 1993 (11.06.93)   | Date of mailing of the international second 01 July 1993 (01.07.   |   |  |  |
|  | pailing address of the ISA/ pean Patent Office   | Authorized officer   |   |  |  |
| Facsimile N                            | o.   | Telephone No.  |   |  |  |

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#### INTERNATIONAL SEARCH REPORT

International application No. PCT/FR 93/00221

| C (Continuat | tion). DOCUMENTS CONSIDERED TO BE RELEVANT   |                            |
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| A            | US,A,5 023 075 (MACCHIO ET AL.) 11 June 1991 see examples                                  | 1,2,4,<br>13-15            |
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# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

FR 9300221 SA 71077

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.

The members are as contained in the European Patent Office EDP file on

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11/06/93

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| US-A-5023075                              | 11-06-91         | None   |   |  |